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Characterization of 36°YX-LiTaO₃ Wafers by Line-Focus-Beam Acoustic Microscopy

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Abstract-Application of line-focus-beam (LFB) acoustic microscopy is extended to characterization of substrates for SHtype SAW devices. Theoretical and experimental studies on a wave mode for characterization are carried out on 36°Y-cut LiTaO₃ wafers. A Rayleigh-type mode of leaky surface acoustic waves (LSAWs) must be employed instead of an SH-type mode of leaky pseudo-surface waves (LPSAWs). Experimental results show that the LSAW propagation should be directed along the X-axis because the LSAW velocities are more sensitive to chemical composition and elastic inhomogeneities. The relations among the LSAW velocities, densities, and Curie temperatures are determined. The LSAW velocity increases linearly at the rate of 0.52 m/s/°C with the Curie temperature. A chemical composition change of 0.03 Li2O-mol%, corresponding to temperature resolution of better than 0.3°C, is easily detected by the velocity measurements. Elastic inhomogeneities due to residual multi-domains, produced during the poling process during wafer fabrication, are interpreted quantitatively by this ultrasonic technology.

I. INTRODUCTION

ERROELECTRIC single crystals of LiNbO₃ and LiTaO₃ are used widely as substrates for SAW devices and for optoelectronic devices. One of the more current important scientific and industrial subjects is to produce and supply high quality large-diameter substrates with homogeneous properties. Much effort has been devoted to the establishment of the crystal growth conditions and wafer fabrication processes, and to the examination and selection of wafers for high quality control of the wafer production process. Wafers for SAW device use are, at present, examined routinely by measuring the Curie temperature, which is directly related to the chemical composition and the SAW velocity, and effectively by observing the chemically etched wafer surface to confirm the complete poling process. Wafers for optoelectronic device use are additionally evaluated by X-ray topography for crystal imperfection and by measuring the optical refractive index. Line-focus-beam (LFB) acoustic microscopy [1] has become available as a very useful and unique method of quantitative characterization of crystals/wafers for such purposes. Characterization is accomplished by measuring the velocity of leaky

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surface acoustic waves (LSAWs), excited on the boundary between a specimen and the water couplant, through V(z)curve analysis. The usefulness of this analytical technique has been demonstrated for characterization of 127.86° rotated Ycut X-propagating (128°YX) LiNbO3 and X-cut 112.2° rotated Y-propagating (X-112°Y) LiTaO₃ wafers for SAW devices concerned with such material problems as congruent chemical compositions and elastic inhomogeneities [2], [3]. Also, it has been demonstrated recently, from the study of optical-use MgO-doped LiNbO₃ crystals [4], that this ultrasonic method has a much greater sensitivity and resolution for other chemical and physical properties, such as chemical composition, density, lattice constant, refractive index, and Curie temperature, than any other systems presently available. It has become possible to make quantitative analyses, by this new type of ultrasonic technology, with such desirable sensitivities in order to resolve present and future materials problems for all the processes associated with the crystal/wafer production.

The application of this ultrasonic method is extended, in this paper, to characterization of the substrates for SH-type SAW devices. Theoretical and experimental studies are carried out to develop a method of material analyses, taking as an example 36° rotated Y-cut X-propagating (36°YX) LiTaO₃ wafers. LSAWs must be employed for characterization instead of leaky SH-type SAWs. The LFB system is applied to study the two scientific and industrial problems of the congruent chemical composition and inhomogeneity occurring in the production line.

II. CHARACTERIZATION WAVE MODE

In LFB acoustic microscopy with perfect directionality, measurements utilize the propagation of leaky waves excited on the boundary between a specimen and the reference liquid, usually distilled water. The method and system are described in detail elsewhere [1]. For application to characterization of piezoelectric single crystals/wafers, we can take advantage of the fact that direct evaluation of the elastic properties, along the desired wave propagation direction, of SAW devices can be made nondestructively in small areas at chosen positions on the specimen. The measurement accuracy is very high: the relative errors for velocity measurements are less than $\pm 0.005\%$ at a selected point and less than $\pm 0.01\%$ over an entire scanning area of 75 mm \times 75 mm [5]. Our previous studies [2], [3] have established a method of characterizing the substrates of LiNbO₃ and LiTaO₃ for Rayleigh-type SAW devices.

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Fig. 1. Calculated results of propagation characteristics of LSAW and LP-SAW: (a) phase velocity; (b) normalized attenuation factor.

Fig. 2. Typical V(z) curve measured for a 36°YX-LiTaO $_3$ wafer at 225 MHz (a) and final spectral distribution obtained by V(z) curve analysis (b).

 36° YX-LiTaO₃ wafers are substrates suitable for the SHtype SAW devices [6]. Before characterizing the wafers by LFB acoustic microscopy, let us consider surface wave propagation in the measurement environment of the water-loaded wafer surface. Theoretical calculations were carried out, according to the analytical procedure of Campbell and Jones [7]. The propagation characteristics, viz., phase velocity, V_L , and normalized attenuation factor, α_L , defined as

$k_L = (\omega/V_L)(1+j\alpha_L)$

where k_L is the complex wave number and ω is the angular frequency, were determined as a function of the wave propagation direction on a 36°Y-cut LiTaO₃ substrate. The results are shown in Fig. 1, using the physical constants of LiTaO₃ published by Warner et al. [8]. The angle of 0° indicates the X-axis propagation on the surface. Two modes could possibly exist, viz., a leaky surface acoustic wave (LSAW), corresponding to the generalized Rayleigh wave, for all directions, and a leaky pseudo-surface acoustic wave (LPSAW), corresponding to the SH-type SAW, for the limited directions 0° to 30° and equivalent directions. The calculated results of the normalized attenuation factor reveal that the LPSAW mode has rather small values, while the LSAW mode has moderate values. A small attenuation factor corresponds to a small wave excitation efficiency at the water/specimen boundary, so that the LPSAW mode is

scarcely excited on the wafer surface by longitudinal waves in the water couplant. The LSAW mode must, therefore, be employed for characterization.

Fig. 2(a) shows a typical V(z) curve for a commercially available 36°YX-LiTaO₃ wafer measured at 225 MHz. From the measurement principle [1], it is easy to understand that only one leaky wave mode contributes to the interference oscillations in this curve. From the spectral distribution as shown in Fig. 2(b), the propagation characteristics that $V_{LSAW} =$ 3127.3 m/s and $\alpha_{LSAW} = 1.11 \times 10^{-2}$ can be obtained by the procedure of V(z) curve analysis [1]. As compared with the theoretical results of Fig. 1, these measured values are very close to those calculated for an LSAW mode, but are quite different from those for an LPSAW mode. The wave mode employed for the measurement is identified as a Rayleigh-type mode of LSAWs, as predicted by the theoretical considerations. We must use an LSAW mode for characterization of 36°Y-cut LiTaO₃ wafers in all the directions.

III. EXPERIMENTS CONDUCTED

Two major problems are investigated here: chemical composition and elastic inhomogeneity. LSAW velocities are measured by the LFB system at 225 MHz. The results obtained are discussed with data of the Curie temperature, measured by differential temperature analysis, and of the density by conventional Archimedes' method, in order to exhibit the interrelations among them.

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	Density	Curie Temperature	LSAW Velocity
Sample No.	ρ (kg/m ³)	Τ _c (°C)	V _{LSAW} [m/s]
1	7471.0	594.1	3122.21
2	7469.0	595.6	3122.76
3	7467.7	598.7	3124.14
4	7465.5	602.5	3126.82
5	7464.5	606.6	3128.43

 TABLE I

 MEASURED PROPERTIES OF 36°YX-LiTaO3 WAFERS WITH DIFFERENT COMPOSITIONS

For this research purposes, 36° Y-cut LiTaO₃ wafers, prepared from crystal boules pulled along the 36° rotated Y axis, are used in the commercially available wafer form of 76.2-mm diam., 0.35 mm thickness with front surface mirror-polished and back surface ground, having the orientation flat perpendicular to the X axis. For the chemical composition study, five wafers were prepared from five crystals grown with different chemical composition ratios. For the detailed inhomogeneity study, four wafers were used with imperfections resulting from undesired conditions during the poling process, but with congruent chemical composition for commercial wafers.

A. Chemical Composition

The Li₂O contents of five wafers increase monotonically from the No. 1 to the No. 5 wafer that is confirmed by the Curie temperatures, as shown in Table I, which were measured after making the following LSAW velocity measurements.

Velocity measurements were made at the center of the wafers by changing the wave propagation direction in one degree steps over 200 degrees. Fig. 3 shows the results of measurements of the angular dependence of the velocities for two wafers of No. 1 and No. 5, for which the chemical composition change was the largest among the five specimens. The results reflect the crystal symmetry of 180°, and the velocities are least in the X-axis propagation direction, which is the direction of greatest interest in 36°Y-cut LiTaO₃ wafers employed for SH-type SAW devices. Due to the different compositions, the velocities for No. 5 are greater than those for No. 1 for all the propagation directions. The two curves exhibit a nearly constant difference over all directions. The angular dependence of the measured velocities is significantly different from that of the calculated velocities for most of the angles. It is considered that the chemical compositions of the specimens used here are different from those used by Warner et al. [8], and it is also suggested that the physical constants



Fig. 3. LSAW velocities for 36° Y-cut LiTaO₃ wafers of No. 1 and No. 5. The dotted line is the calculated curve given in Fig. 1.

measured by Warner et al. [8] were not accurate enough to explain the propagation characteristics obtained here.

Next, to investigate inhomogeneities of the wafer surfaces, LSAW velocities were measured in the X-axis propagation direction by scanning the five wafer surfaces in 1 mm steps over a distance of 64 mm along two diameter lines; parallel and perpendicular to the X axis; shown in Figs. 4 and 5, respectively. Only small velocity variations were detected along both the diameter lines, although relatively large velocity variations, decreasing about 1 m/s in the outer region for the X-axis direction of No. 1 and No. 3 wafers, were observed. Even for the X-axis direction of the No. 1 specimen, the average velocity is 3122.12 m/s, and the maximum difference is 1.66 m/s (0.053%), which is within a tolerance ($\pm 0.04\%$) for SAW substrates [12]. The average velocities increase monotonically from the No. 1 to No. 5 wafer, corresponding to the changes of the chemical compositions.

In order to obtain a quantitative explanation of the velocity changes among the five wafers, the densities, ρ , and the Curie temperatures, T_c , were measured for a crushed powder



Fig. 4. LSAW velocity variations along the X-axis diameter direction of 36° YX-LiTaO₃ wafers with different compositions.

obtained from the central 5 mm \times 5 mm area of each specimen. The average LSAW velocity values for each wafer were determined from the data previously obtained for the two scanning lines, in the corresponding area, for the T_c measurements. These data are listed in Table I.

The maximum deviation of T_c is 12.5°C (2.1%) between the No. 1 and No. 5 wafers. This corresponds to the chemical composition change of 1.3 Li₂O-mol%, from the literature [9]. As the T_c or Li₂O content increases, the V_{LSAW} increases linearly with the maximum deviation of 6.22 m/s (0.199%) and the ρ decreases monotonically with the maximum deviation of 6.5 kg/m³ (0.087%). The LFB system sensitivity to T_c is 1.9°C/(m/s) with the resolution better than 0.3°C, corresponding to 0.03 Li₂O-mol%. For reference, as the Curie temperature for commercial wafers with the effective congruent chemical composition is around 603.5°C, 36°YX-LiTaO₃ wafers have about the LSAW velocity of 3127 m/s and the density of 7465 kg/m³, from Fig. 3.

B. Elastic Inhomogeneity

Serious problems of the elastic inhomogeneities come primarily from chemical composition variations [2], [3], [9]–[14] and secondarily from residual multi-domains [2], [14]. We, now, apply the LFB system to evaluation of several wafers. Unacceptable wafers are usually identified by observing the shift of center frequency and the anomalous frequency characteristics from SAW devices, fabricated on the wafers, in the device production line [14].



Fig. 5. LSAW velocity variations along 90° X-axis diameter direction of 36° YX-LiTaO₃ wafers with different compositions.



Fig. 6. Typical distribution of LSAW velocities measured on a 36° YX-LiTaO₃ wafer.

LSAW velocity distributions were examined for four wafers, obtained from different lots, which produced inferior SAW devices, and which were compared with that for a nominally normal wafer. Fig. 6 shows a two-dimensional velocity distribution for the wafer having the greatest inhomogeneities among the five specimens, when LSAWs propagate in the X-axis direction perpendicular to the orientation flat. The area was sectioned into 6 mm \times 6 mm squares and the LSAW velocities were measured at the center of the squares. The regions of lower velocities can be seen around 3127~3129 m/s located at both right and left hand sides in the outer regions,



Fig. 7. Velocity variations along x- and y-scanning lines of Fig. 6 for a $36^\circ YX\text{-LiTaO}_3$ wafer.



Fig. 8. Typical distribution of LSAW velocities measured on a $36^{\circ}Y\text{-}90^{\circ}X\text{-}LiTaO_3$ wafer.

and the regions of higher velocities at the central parts along the y-scanning line diameter parallel to the orientation flat in Fig. 6. The average velocity is 3130.17 m/s, and the maximum deviation is about 5.07 m/s (0.162%). Fig. 7 shows detailed profiles of the velocity variation along the x- and y-scanning line diameter directions for the X-axis wave propagation over a distance of 64 mm. The velocities along the x-scanning line vary drastically with the maximum deviation of 4.48 m/s, while those along the y-scanning line do so with 2.67 m/s.

In comparison, we attempted to investigate the velocity distributions when the wave propagation direction is changed to the 90° rotated X-axis propagation direction, as shown in Fig. 8 for two-dimensional mapping and in Fig. 9 for the x- and y-scanning lines. As compared with those obtained for the X-axis propagation in Figs. 6 and 7, similar velocity distributions and profiles were obtained by changing the wave propagation direction. However, the maximum deviations of 3.54 m/s (0.113%) for the two-dimensional mapping, 3.59 m/s for the x-scanning line, and 1.29 m/s for the y-scanning line were significantly smaller.

Fig. 10 shows the velocity profiles taken for other three abnormal (A, B, and C), and one normal (D), wafers along the x-scanning lines for the X- and 90° X-axis propagation directions. In comparison with the results for the previous



Fig. 9. Velocity variations along x- and y-scanning lines of Fig. 8 for a $36^\circ Y\text{-}90^\circ X\text{-}LiTaO_3$ wafer.



Fig. 10. Velocity variations along x-scanning line measured for four 36° Y-cut LiTaO₃ wafers. (a): X-axis, (b): 90° X-axis. (A), (B), and (C): for abnormal wafers, (D): for normal wafer.

wafer, small velocity changes were obtained even for the Xaxis propagation as shown in Fig. 10(a). For the 90°X-axis propagation, the variations, shown in Fig. 10(b), still remained smaller to the nearly same level as that of the normal wafer. Setting this inhomogeneity problem aside, we can see clearly that each wafer has a slightly different average LSAW velocity from others for the same propagation direction. This can be explained by the different chemical composition shown in Table I. It is clear from this result that the variation of SAW velocity among wafers, which is one of the most important industrial problems, can be observed accurately with this LFB system for evaluation and sorting.



Fig. 11. LSAW velocities as a function of propagation direction measured for 36° YX-LiTaO₃ wafers with single- and multi-domains.

From this study of the inhomogeneities, we can conclude that LSAWs for the X-axis propagation direction must be employed for complete and accurate characterization of 36° Y-cut LiTaO₃ wafers, because of the available sensitivity.

IV. DISCUSSION

Let us try to explain the observed inhomogeneities. It is easily deduced from the velocity distribution of Fig. 6 that the inhomogeneity was caused by unacceptable poling conditions, as the poling operation was performed with two electrodes formed on the side wall of the crystal boule, at the upper and lower parts shown in Fig. 6. In order to discuss relatively large inhomogeneities, it might be well to consider problems occurring during the poling process, as investigated previously [2]. Fig. 11 shows the experimental results of the angular dependence of the propagation characteristics measured at the center of two specimens of 36°Y-cut LiTaO₃ wafers: one is a normal specimen having a single-domain and the other is a perfectly depoled specimen with multi-domains, prepared by heating the wafer to about 700°C and cooling it without applying voltage. For the normal specimen, the anisotropic curve is similar to the curves in Fig. 3, and the LSAW velocity is 3128.07 m/s in the X-axis propagation direction in Fig. 11, corresponding to the measured T_c of 604.2°C. On the other hand, for the depoled specimen, the curve is a quite interesting one which was not expected. Significant variations between two curves were observed. In particular, the velocity around the X-axis propagation increases remarkably by 10.82 m/s, while the velocity around the 90°X-axis propagation decreases by 9.45 m/s.

With the help of chemical etching techniques, we can observe the distribution of domains from the surface topography made by the rather different etching rates for the positive and negative surfaces. Figs. 12(a) and (b) are photographs taken at the center and at a position of -30 mm from the center, respectively. In Fig. 12(b), a rough surface was observed, corresponding to slow velocities, suggesting that a greater fraction of the area remains unpoled. On the other hand, only a small part of the area in Fig. 12(a) is unpoled, although a perfectly smooth surface can be seen for single-domain wafers. It is thereby confirmed that this wafer had a residual distribution of multi-domains developing in the poling process. When making an interpretation of the velocity



Fig. 12. Micrographs of the wafer surfaces at (a) the center and (b) at a position of -30 mm from the center on X-axis diameter direction in Fig. 6.

distribution and profiles, shown in Figs. 6 and 7, with the information of residual multi-domain distributions obtained from the etched surface and the results of velocity changes in Fig. 11, a contradiction emerges from the assumption that the LSAW velocity increases for the X-axis direction and decreases for the 90°X-axis direction in proportion to the portion of residual multi-domains. That is, the distribution can be explained for the 90°X-axis propagation case, but not for the X-axis propagation case, from the two experimental facts that the velocities decrease monotonically depending on the fraction of multi-domain percent and the velocity for a perfectly depoled area must increase by 10.82 m/s as shown in Fig. 11. This contradiction may be associated with a very interesting scientific problem: differences of elastic properties among single-, multi-, and partial multi-domain LiTaO₃ crystals.

Theoretical calculations show that $36^{\circ}YX$ -LiTaO₃ wafers exhibit very strong piezoelectric characteristics for SH-type SAWs with the electro-mechanical coupling factor K² = 4.54%, while the wafers have very weak piezoelectric characteristics for Rayleigh-type SAWs with K² of 0.00244%. It might, therefore, be expected that the SH-type SAWs for the depoled wafers should exhibit a great change in velocity as the inevitable consequence, and that the Rayleigh-type SAWs should have a small velocity change. This explanation is



Fig. 13. LSAW velocities measured at the two positions of the center and -30 mm away from the center on X-axis diameter line for the 36° Y-cut LiTaO₃ wafer in Fig. 7.

ineffective by the results shown in Fig. 11. The experimental results show that the elastic, piezoelectric, and dielectric constants associated with the Rayleigh-type SAW propagation were changed remarkably by the depoling so that the velocity increased in the X-axis direction and the velocity decreased in the 90°X-axis direction. A clear solution will be obtained by a future study determining the acoustic parameters for a depoled LiTaO₃ crystal by calculating the LSAW propagation characteristics with the data and comparing the experimental results of Fig. 11 with the theory.

Another experimental trial was made to explain the observed inhomogeneities. Angular dependences of the LSAW propagation characteristics were measured at two positions of the center and -30 mm away from the center on the X-axis diameter line in Fig. 7. The results are shown in Fig. 13. The two curves run nearly parallel to each other, similar to the curves of Fig. 3 caused by the different chemical compositions. It is possible to make the inhomogeneities relate to the composition problem. Fig. 14 shows the results of simply obtaining the difference of the two curves of Fig. 13, as a function of the propagation direction. The difference of 4.64 m/s, corresponding to the Curie temperature difference of 8.9°C, in the X-axis suggests the higher Li₂O concentration of 0.91 mol% at the center of wafer than that at the position of -30 mm, using the data of Table I. Such a large change in the composition of a wafer is not an acceptable outcome of the normal crystal growth process. This assumption was experimentally rejected by the Curie temperature measurements having 604.1°C at the center and 603.6°C at the position of -30 mm. It is because the temperature difference of $0.5^{\circ}C$ corresponds to a composition change of 0.05 Li₂O-mol%.

Nevertheless, as regards the inhomogeneities, which were clearly detected by LFB acoustic microscopy, their origins remain unsolved as a materials problem concerned with LiTaO₃ crystals.

V. SUMMARY

Theoretical and experimental studies have been conducted extensively to develop a method of characterization of $LiTaO_3$ and $LiNbO_3$ wafers for SH-type SAW devices, by means of the LFB acoustic microscope system. The applications to investigate the elastic properties of $36^{\circ}YX$ -LiTaO₃ wafers



Fig. 14. Differences of LSAW velocities between the two curves in Fig. 13.

associated with such scientific and industrial problems as chemical composition and inhomogeneities have been demonstrated successfully. A Rayleigh-type LSAW mode, rather than an SH-type LPSAW mode, must be used for measurements because the LPSAW cannot be efficiently excited on the boundary between a specimen and the water coupling liquid. LSAWs propagating in the X-axis direction on 36° Y-cut LiTaO₃ wafers should be employed as the LSAW velocities vary more with compositions and elastic inhomogeneities. This conclusion meets the requirement of characterization of 36° YX-LiTaO₃ wafers developed as substrates for SH-type SAW devices.

The quantitative relations among the LSAW velocities, densities, and chemical compositions have been determined. The LSAW velocity increases linearly and the density decreases monotonically with the Li₂O concentration. It has been shown that the velocity resolution of the system corresponds to a chemical composition change of 0.03 Li₂O-mol%.

The LFB high measurement accuracy system has been shown to be a powerful tool for nondestructive and noncontact detection and evaluation of elastic inhomogeneities of the wafers, caused by the distribution of compositions and residual multi-domains. These result very clearly as two-dimensional distributions and detailed profiles of LSAW velocities which cannot be obtained quantitatively by any other techniques. We have, for the first time, succeeded in providing an interpretation of the relatively large inhomogeneities, produced during the poling process, by measuring the different angular dependences of the propagation characteristics of LSAW velocities between two single- and multi-domain specimens of 36°Y-cut LiTaO3 wafers and the Curie temperatures, and by observing the wafer surface chemically etched with a distribution of residual multi-domains. We have also discovered an unsolved research problem and unexpected differences in elastic properties between the two poled and depoled wafers.

In this study, the characterization method by LFB acoustic microscopy has been successfully completed for substrates of LiTaO₃ and LiNbO₃ suitable for SH-type SAW devices, as well as those for Rayleigh-type SAW devices previously developed [2], [3]. This ultrasonic technology has, therefore, been established as a new analytical technology and can be expected to make a practical contribution to evaluation and establishment of the crystal growth conditions and wafer

fabrication processes of LiTaO₃ and LiNbO₃ crystals for SAW device use and for optoelectronic device use.

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